

EXHIBIT A



Nanocrystalline Silicon Ink

Fabio Zurcher, Brent Ridley, Joerg Rockenberger



Project Weekly Report

Big Picture Objective

- Formulate Ink of Hydrogen-Capped Silicon-Nanocrystals (nc-Si:H)

• [REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

Key Results

- [REDACTED] Amines, Esters, Amides, Polyethers + Anionic Surfactants Good; All Solvents Can Yield “Stable, Milky” Dispersions of nc-Si:H
- Tested 16 Surfactants in Xylene With High-Power Ultrasound: 3 Show Almost Clear, Yellow “Solutions” Of nc-Si:H After 0.5 Micron Filtration. [REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

NC-Si:H - DEVELOPMENT PLAN

STEP	PURPOSE	EXPERIMENTS	OS	Done	
Solvent Screen	1. determine compatibility of nc-Si:H w/ various solvents	1. Ultrasonicate nc-Si:H powder in solvent; check oxidation w/ FTIR	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Surfactant Screen	1. Determine miscibility of surfactants w/ solvents	1. 1% surfactant solution; optical inspection	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Dispersion Screen	1. determine suitability of solvents + surfactants to disperse nc-Si:H	1. Ultrasonicate 1% surfactant solvent solution w/ 0.1% nc-Si:H; Optical inspection + filtration	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Ink Formulation	1. Formulate 5 wt% Si-NC:H ink	1. Refine formulation recipe varying agitation parameters, surfactant conc. etc.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Project Is On Track:

- Dispersion Screen Is Finished
- ☐
- Evaluated 650+ Formulations so far...

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1. The first group of respondents (n = 10) was asked to identify the most important factors influencing their decision to use a mobile phone. The factors were listed in a table, and respondents were asked to rank them in order of importance. The factors were: (1) the need to stay connected, (2) the need to stay informed, (3) the need to stay entertained, (4) the need to stay safe, (5) the need to stay healthy, (6) the need to stay productive, (7) the need to stay social, (8) the need to stay motivated, (9) the need to stay organized, and (10) the need to stay happy.

[REDACTED]

[REDACTED]

[illegible]

Source: *Journal of the American Statistical Association*, 86 (1991), 1033-1042.

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Source: *Journal of the American Statistical Association*, 86 (1991), 1039-1052.

1. *Journal of the American Medical Association*, 1997; 277: 1033-1038.

[illegible]

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Risks Map

Issue	Mitigation Plan	Risk
Agglomerate Size Bigger than 20 nm	<ul style="list-style-type: none"> • Higher Ultrasound Power (smaller tip) • Longer Ultrasonication Times • Increased Surfactant Concentration • Apply Ultrasonication + Surfactant During Etch • Separate Larger Agglomerates By Centrifugation + Filtration • Use Multidentate Surfactants • Let's do photovoltaics... 	H
Very Low Mass Loading (< 0.1 %)	<ul style="list-style-type: none"> • Drop-Casting Instead Of Spin-Coating • Increased Surfactant Concentration • Higher Ultrasound Power (smaller tip) • Longer Ultrasonication Times • Apply Ultrasonication + Surfactant During Etch • Use Multidentate Surfactants 	H
Colloidal Stability	<ul style="list-style-type: none"> • Increased Surfactant Concentration • Use Multidentate Surfactants 	H
Impurity Levels	<ul style="list-style-type: none"> • Keep Surfactant Concentration As Low As Possible • Choose Small-Molecule Surfactants • Oxygen-Free Surfactants 	H
Film Density	<ul style="list-style-type: none"> • Keep Surfactant Concentration As Low As Possible • Choose Small-Molecule Surfactants 	H

3rd Screen: Dispersion Of nc-Si:H

Goals:

- Identify Surfactants + Solvents Which Can Disperse nc-Si:H During Ultrasonication

3. Screening Summary

Surfactants			Solvents									
Name	#	Class	Butyl ether	Xylene	Decaline	Diglyme	Terpineol	Ethyl ethoxy propionate	Anisole	Limonene	DnT acetate	1-Dodecene
hexadecylamine	1	amine										
dimethyldioctylamine	2	amine										
tributylamine	3	amine										
N,N-Diethyldecylamine	39	amine										
Sorbitan monolaurate	4	sorbitol										
Linear polyethyl ester	5	fatty ester										
2-DEB	6	PEO										
Pluronic X-100	7	PEO										
Pluronic X-114	8	PEO										
Pluronic XL-80N	9	PFO										
gepal CO 630	21	PEO										
gepal CO 520	22	PEO										
gepal CO 420	23	PEO										
poly(ethylene glycol) monolaurate	17	PEO										
deep 80	18	sorbit-PEO										
Ardis 130	19	siloxane										
Ardis 900	20	siloxane										
Pluronic SF-135	10	?										
Verpol A	13	?										
Verpol O	14	?										
Rhodoclean EBS	23	?										
K-Sperse 52/MS	27	sulfonate										
K-Sperse XD-A503	29	polymer										
Disstabil KS-873M	31	anionic										
Disstabil 7004	32	?										

☐ = "good" (milky)

☐ = "ok" (turbid or flakey)

☐ = bad (precipitated or reacted)

PEP = poly ethylene oxide
PEP = poly 1-methyl ethylene oxide (propyl oxide)
sorb = sorbitol derivate

Main Conclusions:

- Solvent: everything works ⇒ concentrate on xylene
- Surfactant: amines, PEO, esters, amides + anionic work

nc-Si:H Ink Formulation

Goals:

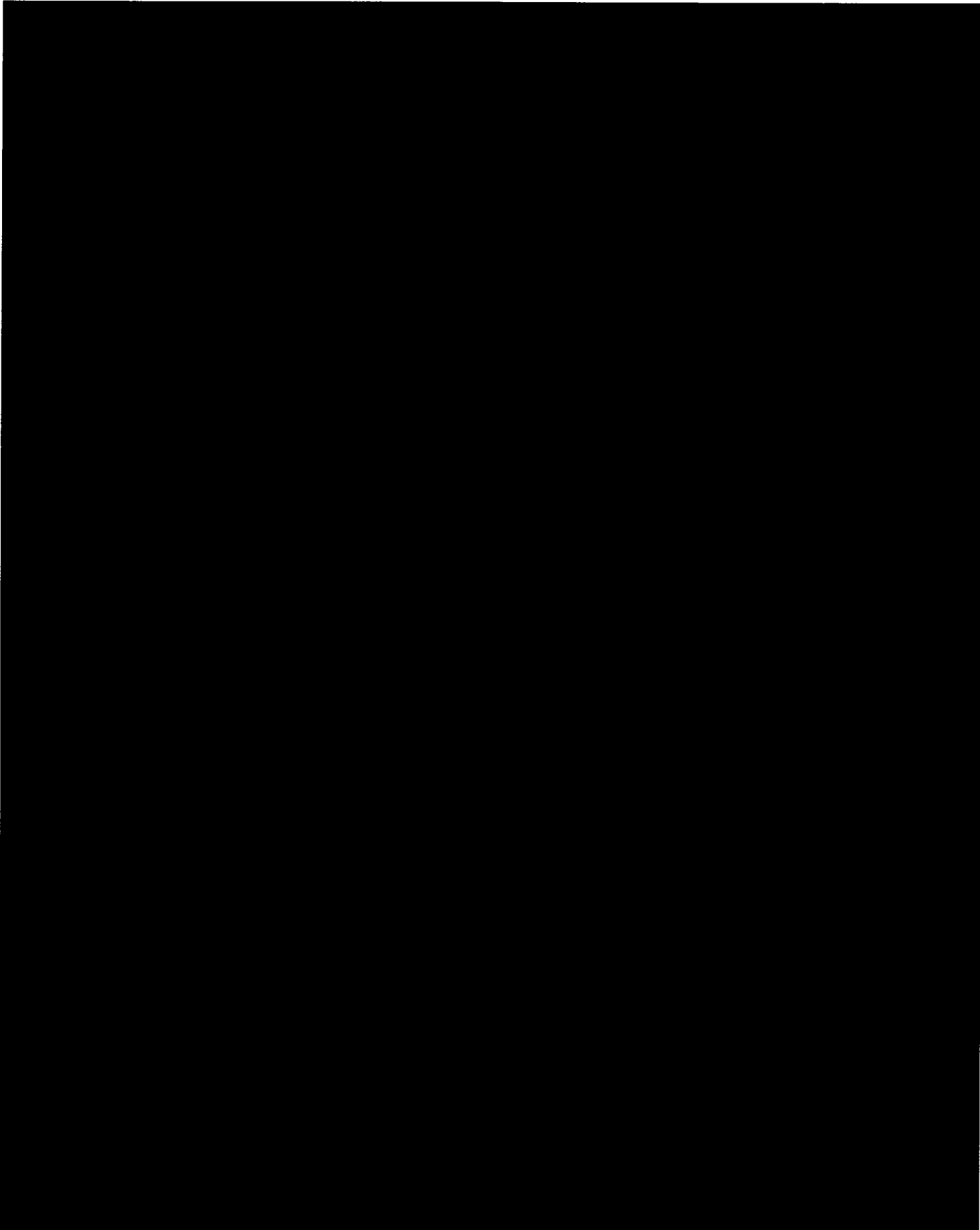
- Identify Surfactant Suitable For Dispersion of <0.5 Micron nc-Si:H Agglomerates In Xylen

Surfactant	After centrifugation	After Filtration	FT-IR Oxidation?
Hexadecylamine		pale yellow	
Dimethyldodecylamine			
Trioctylamine		pale yellow	
4-Dodecyl-diethylenetriamine	yellow SN	yellow	
N,N-Diethyldodecanamide	pale yellow SN	pale yellow	
Sorbitan monooleate		n/a	
Linoleic acid ethyl ester	pale yellow SN		
C12E5		faint yellow	
Triton X-100		faint yellow	
Triton X-114			
Triton XL-80N			
Igepal CO 210			
Igepal CO 520			
Addid 130		n/a	
K-Sperse 152/MS		pale yellow	
Disparlon KS-873N dispersing agent	yellow/orange SN	yellow	
	yellow/orange SN	yellow	

colored, transparent
milky
colored

Main Conclusions:

- 3 Formulations give yellow, mostly transparent solutions after 0.5 micron filtration
- 2 are anionic surfactants!!!! How does that work???
- Si-Mass loading estimated to be ~0.01% ⇒ Surfactant-Si mass ratio: 500



Summary

Results

- All Solvents Can Yield Milky Dispersions of nc-Si:H
- Amines, Polyethers, Esters, Anionic Surfactants “Work”

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

Milestone Weekly Schedule – nc-Si

Crit Path	Milestone	Who	Start	OS	CS	Done ?	Comments
nc-Si	FTIR + TEM of Supernatants	BR/FZ	08/19	08/21			verify identity of supernatants
nc-Si	Sonnication of Surfactants + Solvents as Control	FZ	08/20	08/21			verify identity of supernatants
nc-Si	search for multidentate + polymeric amines	FZ/BR	08/18	08/25			
nc-Si	DOE on sonication: power, time, surfactant concentration	FZ/BR	08/19	08/25			
nc-Si	Screen surfactants in pyrdine	BR	08/20	08/23			

EXHIBIT B



Surface Derivatization of Silicon Nanocrystals

Fabio Zurcher, Brent Ridley



Pros & Cons of the AIBN Reaction

Pros	Cons
<p>Short reaction time (30min)</p> <p>Reliable reaction</p> <p>No obvious source of metal or halogen contamination</p> <p>Reliable and relatively simple isolation/purification step</p> <p>Product is a well defined, dry powder</p> <p>Product is very soluble in hydrocarbons and ethers</p>	<p>Product shows reoxidation</p> <p>Yield is not exceptionally high</p> <p>AIBN byproducts are difficult to remove</p> <p>Product is not extremely soluble in aromatics</p>

Derivatization Reaction Flow

Step	Description
Derivatization	nano-Si stock (etch product suspended in xylene) + dodecene + AIBN + solvent (xylene). [dodecene] = 1M; [Si] = 0.25M; [AIBN] = 0.1M T = 120°C t = 30min
Filtration	Reaction product is filtered hot through a 0.2µm PTFE filter
Precipitation	Product is precipitated with cold MeOH and centrifuged to remove the SN
Wash	The precipitated product is washed with AcN to remove residual AIBN byproducts and then centrifuged
Dry	The remaining solid is dried overnight under Ar or N ₂ flow

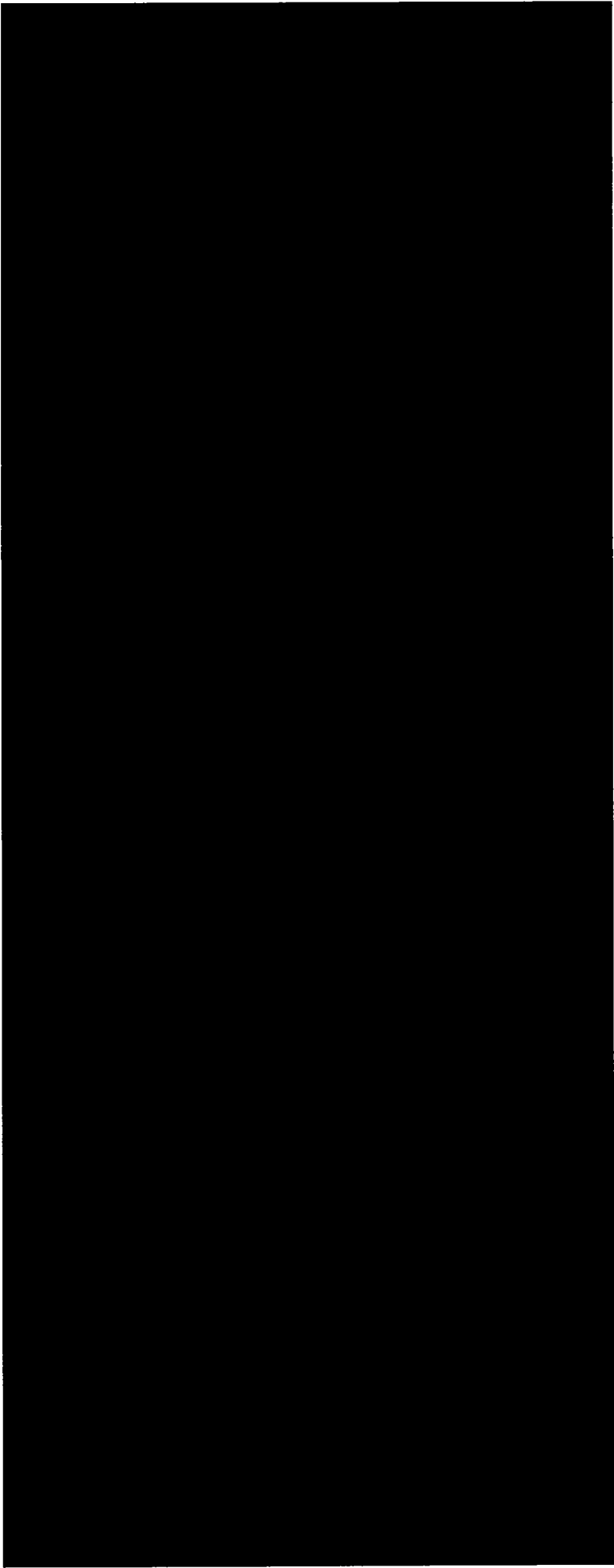
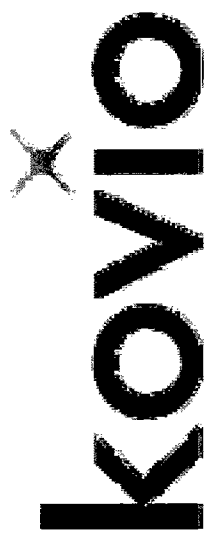


EXHIBIT C



Silicon Film Formation From Nanocrystals

Joerg Rockenberger, Fabio Zurcher, Brent Ridley



Surface-Modified Si Nanoparticles

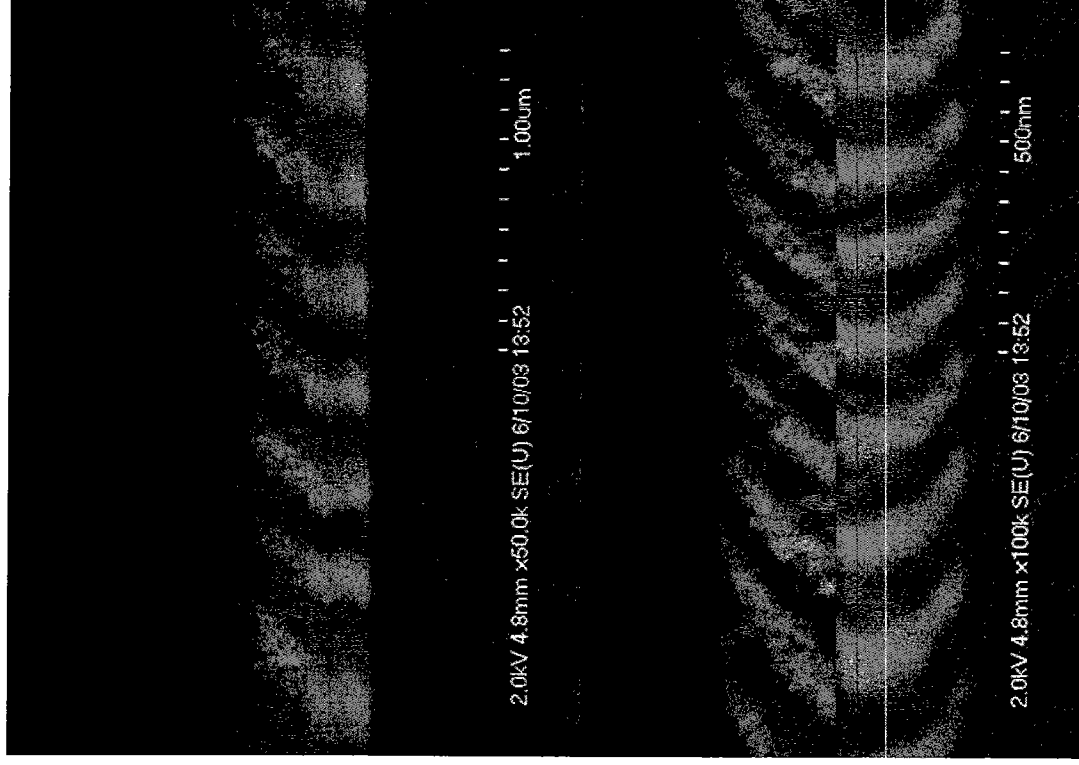
		AIBN	AlEtCl ₂
Synthesis:	Si-NC production [mg/batch]	30	90
	Synthesis + Isolation Time [h]	3	72
	Temperature [C]	120	40
Ink Formulation:	Oxygen – Level [%]	0.5	0.1 – 0.2
	TGA – Mass Loss [%]	20	TBD
	Solubility – Xylene [%]	TBD	5%
	Solubility – Butylether [%]	> 5%	< 1%
Film Characterization:	Oxygen – Level [%]	15 / 11	21
	Carbon - Level [%]	15 / 18	17
	Hydrogen – Level [%]	18	TBD
	SEM – Thickness [nm]	150 – 250	0 - 100
	SEM - Morphology	TBD	very rough – waffle
	XRD – Grain Size [nm]	TBD	TBD
	Tencor – Thickness [nm]	150 -250	85
	Tencor – Roughness [nm]	< 2	6

Biggest Difference: Related to Solubility + Resulting Film Morphology!

Surface-Modified Si Nanoparticles

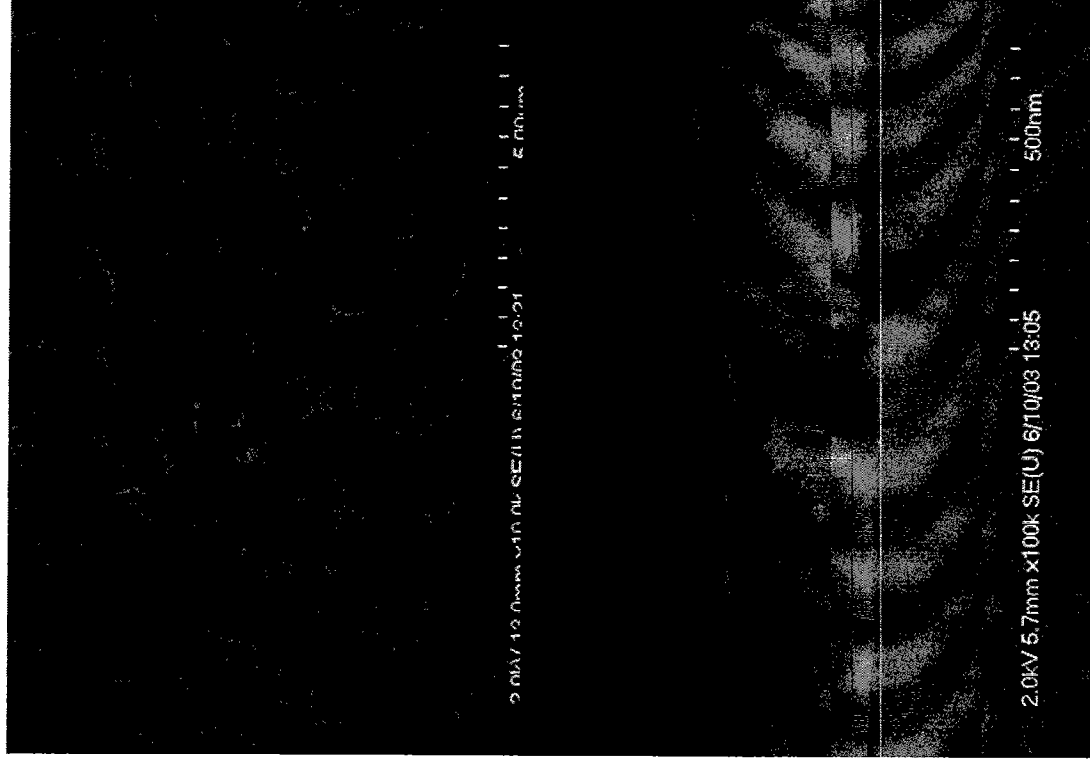
AIBN Reaction

5% in Butylether, 300 rpm
100 C softcure, 900 C hardcure



Lewis Acid Reaction

2% in Xylene, 300 rpm
100 C softcure, 500 C hardcure



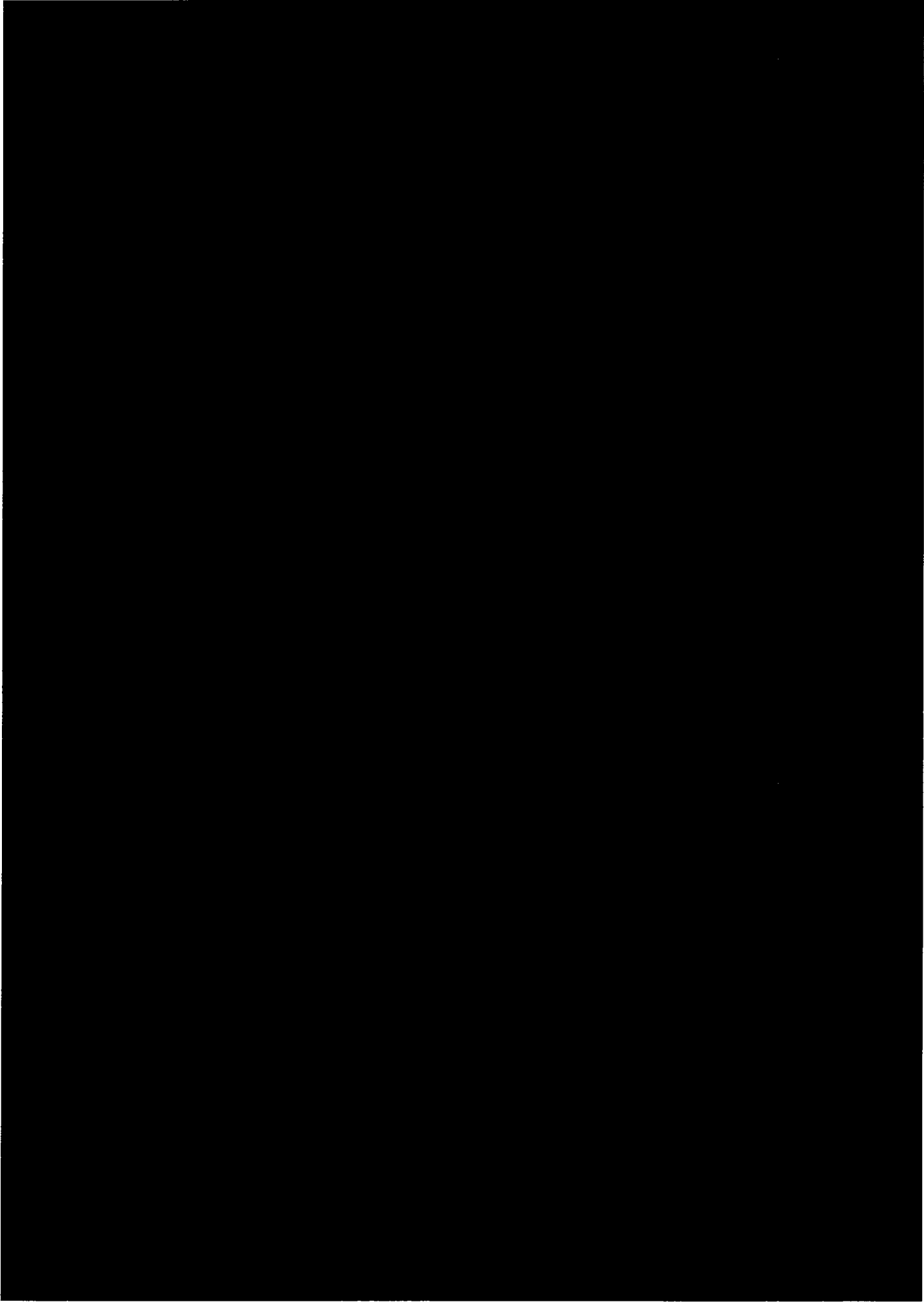
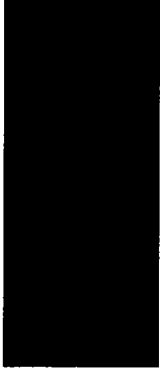


EXHIBIT D

Polysilane ink formulation



Polysilane ink formulation

Goal

- Find solvents that are compatible with cyclosilane.
- Reproducibly provide a polysilane ink formulation with a shelf life of > 1day.

Module	Experiments			
Ink formulation stability	Test cyclosilane compatibility in different solvents			
	<p>2. Thermally polymerize cyclosilane</p> <ul style="list-style-type: none"> – FTIR – Solubility in cyclosilane, cyclooctane <p>3. UV polymerize cyclosilane at RT</p> <ul style="list-style-type: none"> – FTIR – Solubility in cyclosilane, cyclooctane – Weight loss 			

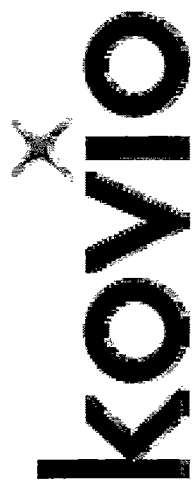
Silane Compatibility With Solvents

Linear Alkanes	Cyclic alkanes	Alcohols/Ketones/Ethers	Aromatics	Ethers	Fluoro
Decane	Cyclohexane	α -Terpineol	Benzene	Dibutylether	Fluorinert FC 70
Tetradecane	Cycloheptane	DHT	Toluene	Butylether	Perfluoro(1-methyl)decane
	Cyclooctane	1-Methoxy-2-propanol	O-xylene	Anisole	Fluorinert FC 70 + Perfluorooctyl sulfonate
	Cyclodecane (tbd)	α -Pinene	Mesitylene		
	Methylcyclohexane	2-Butanone	t-Butyl-toluene		
	t-Butylcyclohexane	2-Heptanone	Cyclohexylbenzene	<div> stable > 2 wks Limited stability white ppt. white ppt., then dissolution two phases </div>	
	cis-decalin	Cyclopentanone	Tetraline		
	trans-decaline	Ethylpyruvate			
	Bis-cyclohexyl	Butylether			

Polysilane ink formulation

Sources that may have an impact on ink stability	Parameters for experimental matrix
Light or temperature induced polymerization	Temperature: ~0C, 45C Light/dark: dark at 0C, clear/amber glass vials at 45C
Solvent purity (water and other impurities)	Distill and dry Cyclooctane, cis-decaline
OH (leaching from glass wall) induced polymerization	Teflon vials and silanized glass vials
Contamination from tips, cap lining	Use current pipettor tips, cap lining
Nature of silane mixture, SiH ₃ groups as radical initiators)	Silane batch: fixed, █21-1A, needed: 30 x 30 uL = 900 uL; NMR control of cyclosilane/cyclohexane ink
Mass loading	Concentration: fixed, 20 vol%, Amount: fixed, 150 uL
Nature of solvent	Solvent: Cyclohexane, Cyclooctane, (Cycloheptane), Ether, toluene, (cyclodecane), decaline
█	█
█	█

EXHIBIT E



Silane Ink Formulation

Solvent Selection and Controlled Polymerization



Solvent/Silane Compatibility

Solvent	bp (°C)	V (cP)	S (mN/m)	Cutoff (nm)	Solubility	FTIR	Thin Film
Cyclooctane	151	1.0			OK	No Si-O	Alkanes/Aromatics
cis-Decalin	193	3.0			OK	No Si-O (100 °C)	UV Film OK
Decalin (mixture)	191	2-3			OK		
o-Xylene	144	0.8	29.5		OK	No Si-O (100 °C)	
Tetralin	207	2.1			OK		
Methylnaphthalene	240	3.1			OK	No Si-O (150 °C)	
Tetradecane	252	2.1			Cloudy→OK	No Si-O (150 °C)	
D4-Cyclomethicone		2.4	17.4		OK	No Si-O (150 °C)	Exotics
D5-Cyclomethicone		3.8	17.4		OK	No Si-O (150 °C)	
Cineole*	176	2.3			OK→Solids	No Si-O (100 °C)	Ethers/Alcohols
EG-dibutylether	203	14			OK→Solids		
3-Octanol*	174	7			Cloudy	Oxidation	
2-Ethylhexanol	182	4-6			Cloudy	Oxidation	
Dihydroterpineol*	208	46			OK	Oxidation	
Dihydroterpineol (IEF)	208	46			OK→Solids		
Terpinen-4-ol*	212	12			OK	No Si-O (150 °C)	Film oxidation
Terpineol*	217	37	32		OK	No Si-O (150 °C)	Film oxidation
Pine Oil 60		5			OK	Oxidation	

*Similar results after drying solvent over molecular sieves

Silane Polymerization

Sample	Loading (vol%)	Solvent	Exposure (min)	Observation (solution)	Observation (cast)	FTIR
UV			UV			
4-60-0	none	c-C ₈ H ₁₆	5min	Clear	N/A	N/A
4-60-1	5%	c-C ₈ H ₁₆	5min	Cloudy (from walls)	Good wetting/film	No change(s)
4-60-2	5%	o-xylene	5min	Clear (even walls)	Good wetting/film	No change(s)
4-60-3	5%	c-C ₈ H ₁₆	20min	Clear	Good wetting/film	No change(s)
4-62-1	25%	c-C ₈ H ₁₆	20min	Clear	Good wetting/film	No change(s)
4-62-2	25%	c-C ₈ H ₁₆	60min	Milk	N/A	Broad, Baseline
4-63-1	25%	o-xylene	40min	Cloudy	Good wetting/film	Broadening
4-68-1	100%	none	20min	Clear, viscous		Broadening
4-68-2	100%	none	60min	Pale amber, viscous		
Molecular Sieve			Sieves			
4-58-1	5%	c-C ₈ H ₁₆	none	Clear	Poor wetting/film	N/A
4-60-2N	5%	o-xylene	none	Clear	Poor wetting/film	N/A
4-58-1S	5%	c-C ₈ H ₁₆	4days	Clear	Poor wetting/film	No change(s)

White films form routinely form on walls above liquid level (from vapor)

Viscous Silane(s) Solubility

[illegible]

*** 10% solutions – all others 20%**

Silane Ink Formulation

Big Picture Objective:

- Formulate silane ink suitable for inkjet printing (viscosity, surface tension, etc.)
 1. Appropriate solvent selection
 2. Controlled silane polymerization
- [REDACTED]
- Investigate alternatives to inkjet technology (microspot...)



Key Results:

- The viscous tertiary alcohol terpinen-4-ol is retained in UV-spun silane films and leads to oxygen and carbon loaded silane films after curing at 400 °C
- After drying over sieves, alcohol and ether solvents still cause problems with solubility and/or oxidation – so far, no solvents containing oxygen have worked except for the cyclomethicones
- UV polymerization reactions initiated – control of viscosity is underway [REDACTED] solvents [REDACTED], but polymerization (and precipitation) occurs in both cyclooctane and xylene

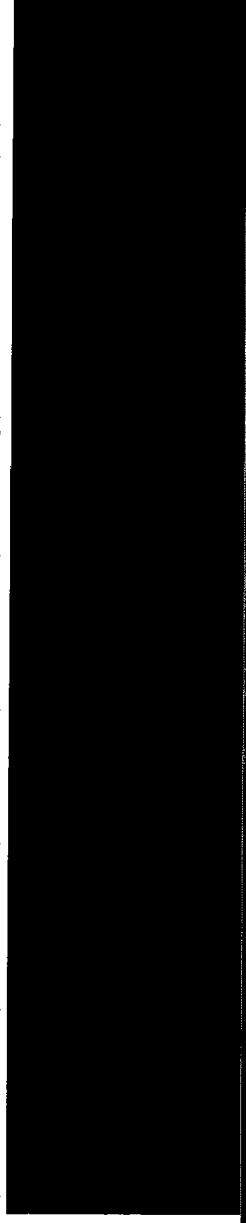


EXHIBIT F



Solvent Compatibility Tables

Technical Report

[REDACTED]

[REDACTED]

Activated Alumina Purification for Solvent Screen - ■■■

Purpose

- Purify inkjet solvent candidates and test solubility, stability, and printability

Method

- Single-pass column purification of solvent – 9g activated alumina (500 °C under vacuum 6h), collecting 4mL fractions in amber vials after discarding first mL
- 20% solution using ■■■ SOP polysilane

Solvents

- Alkanes: Tetradecane, dicyclohexyl, decane, pinane, *t*-butylcyclohexane, isopropylcyclohexane, trimethylcyclohexane
- Aromatic: Methylnaphthalene, tetralin, cyclohexylbenzene
- Ethers: Diethylene glycol diethyl ether, dibutyl ether
- Halogenated: Chlorooctane, cyclohexyl chloride
- Silane: Tetraethylsilane

Analysis Proposal

- Suggested testing, in order of execution: miscibility and solubility at 50% and 20%, stability at 20%, viscosity, contact angle, printability, NMR, GC-MS

Results

- All 15 solvents were poor solvents for polysilane!
- Most were turbid solutions or two phase mixtures
- Some solvents formed two clear phases – tetradecane, decane
- What makes *cis*-decalin and cyclooctane (and cyclodecane) so good?

Solvent/Silane Compatibility - 2004

Solvent	bp (°C)	η (cP)	γ (mN/m)	Solubility (5-10 vol%)	FTIR	Thin Film
Alkanes						
Cyclooctane	151	2.2		OK	No Si-O	TFTs
Nonane	151	0.6	25	OK→Precip		
Decane	174	0.8	23	OK→Precip		
Decalin (mixture)	191	2.2	32	OK		
cis-Decalin	193	3.0	32	OK	No Si-O (100 °C)	TFTs
Cyclodecane	201	4.3		OK	No Si-O (150 °C)	
Dicyclohexyl	207	3.3		OK*		
Dodecane	216	1.2	25	OK→Precip		
Tetradecane	252	1.9		Cloudy→OK *	No Si-O (150 °C)	
o-Xylene	144	0.8	30	OK*	No Si-O (100 °C)	
1,2 Dichlorobenzene	180	1.3	37	Cloudy→OK *	No Si-O (150 °C)	
Tetralin	207	2.1	33	OK*	No Si-O (100 °C)	
Methylnaphthalene	240	3.1	40	OK*	No Si-O (150 °C)	UV Film Patchy Streaks
Aromatics						
2,6-Lutidine	144		32			
Quinoline			43	OK	Si-O (150 °C)	
Anisole	154		36	Cloudy (10%)	No Si-O (150 °C)	
Phenylether	256		39	Cloudy (10%)	No Si-O (150 °C)	
D4 Cyclomethicone		2.4	17	OK	No Si-O (150 °C)	
D5 Cyclomethicone		3.8	17	OK	No Si-O (150 °C)	UV Film Patchy Streaks
Heteroatomic						

* Limited solubility above 5% or with polymeric silanes

Italicized viscosity values are from literature, not in-house measurements

Solvent/Silane Compatibility (Problematic) - 2004

	Solvent	bp (°C)	η (cP)	γ (mN/m)	Solubility (5 vol%)	FTIR	Thin Film
Exotics	D4 Cyclomethicone		2.4	17.4	OK	No Si-O (150 °C)	
	D5 Cyclomethicone		3.8	17.4	OK	No Si-O (150 °C)	UV Film Patchy Streaks
Ethers/Alcohols	Cineole*	176	2.3		OK \rightarrow Solids	No Si-O (100 °C)	
	EG-dibutylether	203	14		OK \rightarrow Solids		
	3-Octanol*	174	7		Cloudy	Oxidation	
	2-Ethylhexanol	182	4-6		Cloudy	Oxidation	
	Dihydroterpineol	208	46		OK	Oxidation	
	Dihydroterpineol (IFF)	208	46		OK \rightarrow Solids		
	Terpinen-4-ol*	212	12		OK	No Si-O (150 °C)	Film oxidation
	Terpineol*	217	37	32	OK	No Si-O (150 °C)	Film oxidation
	Pine Oil 60		5		OK	Oxidation	

*Similar results after drying solvent over molecular sieves